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Microwave Aquametry: An Effective Tool for Nondestructive Moisture Sensing

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Moisture content in solid, granular and pulverized materials is one of the most important material parameters during production, trading, processing and storage of those materials. Recent advances in application of microwave measuring techniques to nondestructive determination of moisture content are reviewed, with a special emphasis being put on a newly developed concept of a density-independent calibration. It is concluded that those techniques provide accurate, fast and nondestructive means for moisture content testing in such materials and satisfy requirements of automated industrial processes, scientific laboratories, material mass storage, personnel safety and long-term transport.

Key Words. Moisture content, measurements, microwave radiation, electromagnetic waves.

1. Introduction

The term ''aquametry'' is used here as a synonym of: ''measurement of moisture content in solid and liquid materials,'' analogous to ''hygrometry'' which is a well established branch of metrology devoted to ''measurement of water vapor content in gases, mainly in air.'' The adjective ''microwave'' in the title of this paper indicates that it will be concerned with moisture content measurement of solids and liquids using methods and instrumentation derived from classical microwave techniques (resonant cavities, waveguide, transmission line, free-space measurements). The subject of interest for microwave aquametry is searching solids of different form and structure as well as liquids containing water for identification of their properties when placed in electromagnetic fields of radio and microwave frequencies (attenuation, reflection, phase angle, shift of resonant frequency,

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etc.). In this aspect microwave aquametry utilizes some physical theories on dielectric mixtures and bound water. But microwave aquametry has also strictly defined practical objectives, namely quantitative measurements of water content in materials, which are important from an economic point of view. Since water occurs in most materials in nature as a natural component of the material or is introduced during technological processes, it is quite obvious that measurement and control of moisture content have great economic and technical importance. Domination of the practical aims over the cognitive purposes influenced the development of microwave aquametry in the past and have also a serious impact on its present state. The purpose of this paper is to present in more detail some of the recent developments in the field, intending to sketch the picture rather than paint it accurately and showing all its colors and complicated components.

Typical nondestructive techniques for determining moisture content in material consist of measuring the electrical properties of the material in a sample holder and relating these properties to the moisture content. These techniques have their roots at the beginning of the twentieth century when the possibility of rapid determination of moisture content in grain by measuring the dc resistance between two metal electrodes inserted into the grain sample was established [1]. This resistance was found to vary with moisture content. Later, samples of wet materials were placed in the path of an electromagnetic wave between two horn antennas and the simple relationship between the propagation constant and the amount of water was easily determined. Both the simplicity of the measuring arrangement and practical utility of the results were fascinating. Because of the particular properties of microwave radiation, (frequencies between 1 GHz and 100 GHz), the new method appeared to surpass all other previous methods for measuring moisture content in solids, such as chemical methods, methods using radiofrequencies (several MHz), infrared and ionizing radiation. The following advantages were obvious since the early experiments:

(a) contrary to lower frequencies, the dc conductivity effects on material properties can be neglected;
(b) penetration depth is much larger than that of infrared radiation and permits the probing of a significant volume of material being transported on a conveyor or in a pipe;
(c) physical contact between the equipment and the material under test is not required, allowing on-line continuous and remote moisture sensing;
(d) in contrast to infrared radiation, it is relatively insensitive to environmental conditions, thus dust and water vapor in industrial facilities do not affect the measurement;

(e) in contrast to ionizing radiation, microwave methods are much safer and very fast;
(f) water reacts specifically with certain frequencies in the microwave region (relaxation) allowing even small amounts of water to be detected;
(g) contrary to chemical methods, it does not alter or contaminate the test material, thus the measurement is nondestructive.

These features combined with great potential savings in fuel, energy, manpower and improvement of the quality of products resulting from the application of moisture content measurement and control, created a powerful incentive for research and equipment development all over the world.

In the mid-sixties several manufacturers of microwave moisture meters were established on the market. Among them were SCANPRO[1]AB in Sweden, AEI and RANK Precision Industries Ltd. in Great Britain, UNIPAN/WILMER in Poland, KAY-RAY in the U.S. (who expanded its line of nuclear radiation meters to include microwave instruments), and COMPUR AG in Germany (who produced meters based on research by Bayer AG). There were new companies being created and old ones bought by others; successful projects were developed and others closed and forgotten; but fascination with the potential of the technology has lasted with varying intensity to this day. The state of knowledge on the subject was summarized several times during the years [2–6]. The first meeting devoted to the exchange of ideas on the subject took place in 1980. The list of papers published at that time exceeded 400 [5]. Professional meetings took place more frequently in the late eighties and a tradition of annual meetings (Feuchtetage) was established in Germany [7,8]. Later another meeting of more international character was established, namely the IEEE International Microwave Symposium Workshop on Electromagnetic Wave Interaction with Water and Moist Substances in 1993 in Atlanta [9], in 1996 in San Francisco [10,12], in 1999 in Athens, Georgia [11], and recently in Weimar, Germany [13].

The total number of microwave moisture meters manufactured during the last forty years throughout the world is unknown. The total investment in research on the adaptation of microwave techniques to aquametric purposes and the number of unsuccessful projects also remain unknown. But the bibliography of the subject, on both physical background and practical application contains well over one thousand entries [5,8,9,12] and does not cover internal reports of proprietary character nor contributions to closed or semiclosed conferences and seminars, (the full text of which were not published in the generally accessible literature). A recent survey indicated existence of over thirty companies involved in manufacturing and

applying moisture meters based on the measurement of microwave parameters [12]. Eleven countries on three continents are represented and materials involved extend from sand to paper and from grain and soil to living fish. This list provides evidence that microwave aquametry is not only a subject of academic discussions and dissertations, but also an accepted tool in the field of nondestructive moisture monitoring and control in modern factories and laboratories.

2. Principles and Definitions

The moisture content of material may be defined on a wet basis (w.b.) as a ratio of the mass of water, m_w, to the mass of the moist material, m_m,

$$\xi = \frac{m_w}{m_m} = \frac{m_w}{m_w + m_d} \tag{1}$$

or, on a dry basis (d.b.), as a ratio of the mass of water in the material to the mass of dry material, m_d,

$$\eta = \frac{m_w}{m_d} = \frac{m_m - m_d}{m_d} \tag{2}$$

Most often the quantities ξ and η are expressed in percentage. The definition expressed by Eq. (1) is most frequently used in practice, and when the concept of moisture content is related to a certain volume of material, v, it can be rewritten as follows:

$$\xi = \frac{m_w/v}{m_w/v + m_d/v} = \frac{k}{k+g} = \frac{k}{\rho} \tag{3}$$

where k is the partial density of water, g is the partial density of dry material, and ρ is the density of moist material. Other relationships resulting from Eqs. (1)–(3) are:

$$\begin{aligned} &\xi = \frac{\eta}{1+\eta}, \qquad \eta = \frac{\xi}{1-\xi}, \qquad k = \frac{m_w}{v} = \rho\xi \\ &g = \frac{m_d}{v} = \rho(1-\xi) = \frac{\rho}{1+\eta} \end{aligned} \tag{4}$$

There are many parameters of materials that can be correlated with the density of water in the material, k, but from Eq. (3) it is obvious that fluctuations in the material density, ρ, have as much influence on moisture content as the variation in k. This observation is universal, because this

disturbing effect of material density does not depend on the electrical method applied for moisture content determination. Thus, when k is determined from electrical measurement, determination of moisture content from Eqs. (1) or (3) requires that ρ be known. This information can be obtained by keeping the mass of moist material in the measuring space constant during the calibration as well as during the measuring procedure; or by performing separate density measurements, for example by weighing a sample of given volume, or by using γ-ray density gauge. A third approach is to use a *density-independent* function, an expression relating the moisture content with electrical properties of the material independent of density and to eliminate, or seriously limit, the density effect in moisture content measurements. It might be interesting to note that in many cases the dry basis moisture content is linearly related to the measured electromagnetic quantities, while the wet basis moisture content exhibits quite nonlinear relationship. In such cases it is wise to calibrate the system for dry basis moisture content and then transform the results of measurement to the moisture content on the wet basis required in many branches of industry.

Standard methods of moisture content determination are *direct* methods, based on the definitions of Eqs. (1) or (2) and performed in laboratories according to procedures described in formal documents of national or international character. The most often used method involves weighing a sample of moist material, removing water by evaporation and reweighing the remaining dry material; another (the Karl Fischer method) uses extraction and chemical titration. The whole procedure is precisely described, giving time and temperature of drying, exact amount of chemicals to be used, etc. These methods are accurate but do not provide rapid results. Drying for up to three days is required in some cases. For rapid moisture content determination and monitoring, indirect methods calibrated against the standard methods have been used, and the method using measurement of microwave properties of moist material is one of them.

Interaction of an electromagnetic wave with moist material can be expressed in terms of the complex value of the propagation constant of the wave in a dielectric medium as

$$\gamma = \alpha + j\beta = j\frac{2\pi}{\lambda}\sqrt{\varepsilon - p} \tag{5}$$

where $\varepsilon = \varepsilon' - j\varepsilon''$ is the relative permittivity of the medium, where ε' is the dielectric constant and ε'' is the loss factor, and $p = (\lambda/\lambda_c)^2$, where λ and λ_c denote free-space and waveguide cut-off wavelengths, respectively. Eq. (5) may be solved for two components of the propagation constant being expressed as:

for the attenuation constant

$$\alpha = \frac{2\pi}{\lambda}\sqrt{\frac{\varepsilon' - p}{2}\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon' - p}\right)^2} - 1} \quad [\text{Np/m}]$$

for the phase constant

$$\beta = \frac{2\pi}{\lambda}\sqrt{\frac{\varepsilon' - p}{2}\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon' - p}\right)^2} + 1} \quad [\text{rad/m,}] \tag{6}$$

In free space, where $p = 0$, the following approximate expressions can be used to relate the electromagnetic wave propagation to the properties of moist materials, assuming that $\varepsilon'^2 \gg \varepsilon''^2$ which is valid in most practical situations,

$$\alpha \simeq \frac{\pi}{\lambda}\frac{\varepsilon''}{\sqrt{\varepsilon'}}, \qquad \beta \simeq \frac{2\pi}{\lambda}\sqrt{\varepsilon'} \quad \text{and} \quad |\Gamma| \simeq \frac{\sqrt{\varepsilon'} - 1}{\sqrt{\varepsilon'} + 1} \tag{7}$$

where Γ is the voltage reflection coefficient from the surface of the moist material. Thus, by measuring the more practical quantities,

$$A = 20\log|\tau| = 8.686\alpha d \quad [\text{dB}]$$

and

$$\phi = (\beta - \beta_0)d = \frac{2\pi}{\lambda}(\sqrt{\varepsilon'} - 1) + n360 \quad [\text{deg}] \tag{8}$$

where A is the attenuation of the material sample in decibels and ϕ is the phase shift in degrees; β_0 is the phase constant in free space; n is an integer to be determined when the thickness d of the material layer is greater than the wavelength in the material, and the transmission coefficient $|\tau| = \exp(-\alpha d)$. The integer n can be found by repeating the measurement with samples of different thickness or by taking the measurements at two frequencies [14]. It is clear from the above that the parameters of the electromagnetic wave are affected by the material relative permittivity which in turn is related to the water content in the material. It is true, however, that the relative permittivity also depends on material temperature, density, shape and dimensions of its particles, chemical composition, etc. [15]. This is where the real troubles start and the science of aquametry begins.

3. Instrumentation

All instruments manufactured recently contain modern microwave integrated circuitry, high speed signal processors and efficient power supplies, and are equipped with modern microcomputers (often operating on the Windows platform), with modems, high capacity data storage and other gadgets typical of modern measuring instruments. Because of general progress in microwave integrated circuit (IC) technology during recent years, the price of microwave components has recently been quite comparable with the price of components (mixers, amplifiers, oscillators, filters, etc.) operating at much lower frequencies. This is another advantage, since very often in the past, application of microwave meters was restrained because of their higher costs. In addition to the progress which has been typical for other measuring instruments, there are certain developments and recent enhancements specific for microwave moisture meters. Some of them are briefly reviewed below.

3.1. Metrological Enhancements

It may be observed from Eq. (7) that the components of the propagation constant, α and β, are dependent upon the relative permittivity of the moist material. Since the relative permittivity in turn depends on moisture content ξ, density ρ, and temperature τ, the components can be written in a general form:

$$\alpha = \psi_1(\xi, \rho, T) \quad \text{and} \quad \beta = \psi_2(\xi, \rho, T) \tag{9}$$

According to the definition of Eq. (3) and expression in Eqs. (8) and (9), the components of the propagation constant can be easily expressed in terms of measured variables A and ϕ, as

$$A = \Phi_1(k, g, T) \quad \text{and} \quad \phi = \Phi_2(k, g, T) \tag{10}$$

These two equations can be solved to express the partial densities of water and dry material in terms of measured variables:

$$k = \Psi_1(A, \phi, T) \quad \text{and} \quad g = \Psi_2(A, \phi, T) \tag{11}$$

In general, this operation known as an inverse problem, can be very complex and uncertain, but in the case of moisture content in most materials, it can be quite simple. Thus, the moisture content can now be expressed as:

$$\xi = \frac{\Psi_1(\alpha, \phi, T)}{\Psi_1(A, \phi\ T) + \Psi_2(A, \phi, T)} \tag{12}$$

which contains only the wave variables, A and ϕ, and temperature T, determined experimentally. Also the density of the wet material

$$\rho = \Psi_1(A, \phi, T) + \Psi_2(A, \phi, T) \tag{13}$$

can be determined at the same time. Thus, the density of moist material is no longer a disturbing factor in the moisture content measurement, but it can be determined during this measurement and used for other purposes in a technological process. Identification of the relationships (12) and (13) is called *the calibration* of the measuring system. It has been suggested recently [16] that carrying out the measurements of two wave parameters at two different frequencies should allow determination of *four variables*, for example, moisture content ξ, bulk density ρ, temperature of the material T and material layer thickness d. Selection of two appropriate frequencies remains to be determined for any given material.

In a search for more efficient and accurate ways of instrument calibration, other approaches were also explored. Artificial neural networks seem to be especially useful [17]. An artificial neural network is a collection of simple interconnected analog signal processors, providing a mathematical structure that can be trained to map a set of inputs to a set of outputs. The inputs are the measured data of A and ϕ, and the output is the value of moisture content, ξ. For experimental data taken in free space at eight frequencies between 10.3 GHz and 18.0 GHz for wheat in the moisture content range from 10% to 19% and at temperatures between –1°C and 42°C [18], the standard error of calibration was 0.135% moisture. When the network was trained using only the amplitude of the transmission coefficient measurements as the inputs, the value increased to 0.219%. This is an important observation, because eliminating the need for the phase measurements greatly reduces the complexity of the hardware required to make the measurements. Application of principal component analysis [19] and partial least-squares regression [20] to the same set of experimental data provided standard errors of performance, of 0.232% and 0.210%, respectively. One should keep in mind, however, that the average spread of moisture content in triplicate 10-gram samples determined by the standard oven method for wheat (130°C for 19 hr) was 0.176% moisture with standard deviation of 0.077% moisture [21]. The interesting conclusion of the last study is that for the two-parameter measuring system, using more than one operating frequency does not improve the predicted accuracy of moisture content measurement.

For some time now, the concept of a density-independent function has been considered a vital way of limiting the density effect in moisture content measurement [22]. The idea is to find an expression or function, X, which can be correlated with moisture content, ξ, in a form

$$\xi = a_1 + b\,X \quad \text{or} \quad \xi = a_2 + b_2\sqrt{X} + b_3 X \tag{14}$$

providing elimination or serious limitation of the density effect. Originally, the ratio of the two measured quantities, A and ϕ, was considered to be such a function. Later, a permittivity related function was proposed [23,24] in the form

$$X = \varepsilon''/(\varepsilon' - 1) \tag{15}$$

It has been shown [25], that Eq. (15) is a part of the original ratio which in turn is a linear function of the material loss tangent and can be expressed as

$$A/\phi = \mathbf{c} \tan \delta \sqrt{\varepsilon'}/(\sqrt{\varepsilon'} - 1) \tag{16}$$

where $\tan \delta = \varepsilon''/\varepsilon'$, and $\mathbf{c}$ is a constant. Because both measured variables of Eq. (8) are directly proportional to the material layer thickness, the density-independent function, Eq. (16), can be correlated with the material moisture content without regard to fluctuations in the material layer thickness d. This is often a valuable feature.

A recently established density-independent function [26] is based on the observation that in the complex plane, the normalized variables ε'/ρ and ε''/ρ for all temperatures and moisture contents can be expressed by the linear equation

$$\varepsilon''/\rho = a_f(\varepsilon'/\rho - b_0) \tag{17}$$

where a_f is the slope of the line, which depends only upon the operating frequency, and b_0 is the intercept constant, which, for a given material, has the same value at all frequencies and corresponds to the density-normalized zero-moisture material permittivity or to the density-normalized permittivity of the material at very low temperature. As an example, Figure 1 shows such a diagram, sometimes called an Argand diagram, for wheat data discussed above [18]. The density of the material can be calculated from Eq. (17); thus, the procedure allows simultaneous determination of the material density and moisture content. The density-independent function for moisture determination can be written as

$$X = \sqrt{\frac{\tan \delta}{\rho}} = \sqrt{\frac{a_f b_0 \tan \delta}{a_f \varepsilon' - \varepsilon''}} \tag{18}$$

The interesting feature of the above relationship is that as more and more experimental data for grain have become available (for corn, soybeans, oats, etc.), all variables in Eq. (18) have been found to have similar values, and since at a given frequency $a_f b_0$ is a constant, one can consider it a *universal*

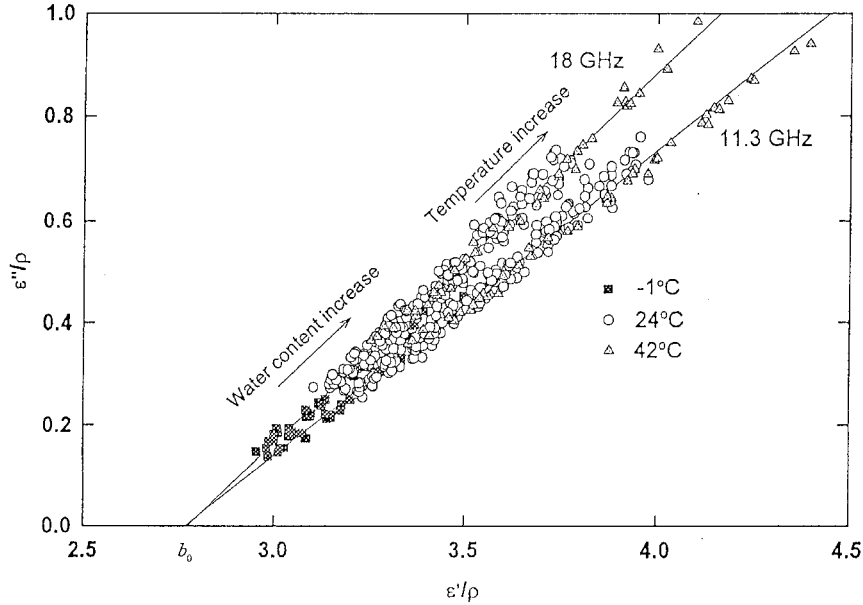

Figure 1. Argand diagram of the normalized effective relative permittivity for wheat at indicated frequencies and temperatures.

function. It must be stressed that the mentioned commodities have pronounced differences in kernel dimensions, shape, bulk densities and composition, and the potential for a common calibration equation for moisture content determination in all of them should motivate further research.

3.2. New Sensors and Transducers

There have been recent developments in microwave moisture sensing devices related to progress achieved in flat, microstrip, patch antennas [28–30], as well as to wide application of microwave resonators (cavity and microstrip) [31,32]. Sensors based on the principle of the reflected wave measurements are easy to use as they allow one-sided sensing and their robust construction permits operation at the bottom of concrete mixers, in walls of chutes, etc. Several open-ended sensors are shown in Figure 2 together with their simplified equivalent circuit. Theory related to the operation of the conical-tip open-ended probe was presented [33], as well as a comprehensive study of various probe types [34]. The input admittance of the sensor is related to the permittivity of the material in which the line is

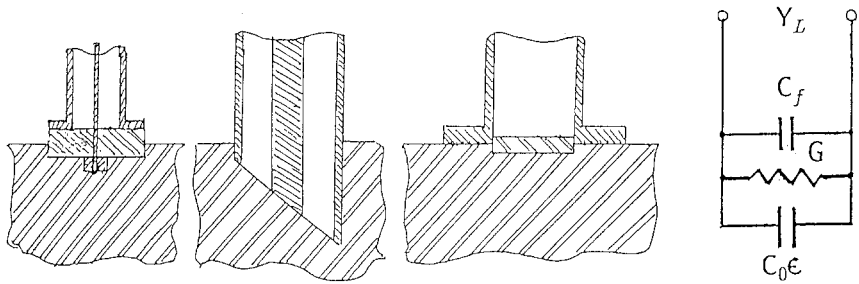

Figure 2. Various open-ended transmission line structures used as sensors in microwave moisture content measurements. From the left, microstrip line, coaxial line, cylindrical waveguide and their simple equivalent circuit.

immersed, and, in turn, the reflection coefficient is a function of the admittance expressed as:

$$\Gamma = \frac{1 - Y_L}{1 + Y_L} \quad \text{where} \quad Y_L = G + j\omega(C_f + \varepsilon C_0) \tag{19}$$

and G is the conductance and C_f and C_0 are capacitances as shown in Figure 2. One example of a reflection sensor is a microstrip open-ended probe used for moisture sensing in the production of curd cheese [35]. Changes of moisture content produce changes in the resonant frequency, as shown in Figure 3, where the reflection loss (Γ expressed in decibels as $R_L = -20 \log |\Gamma|$) is

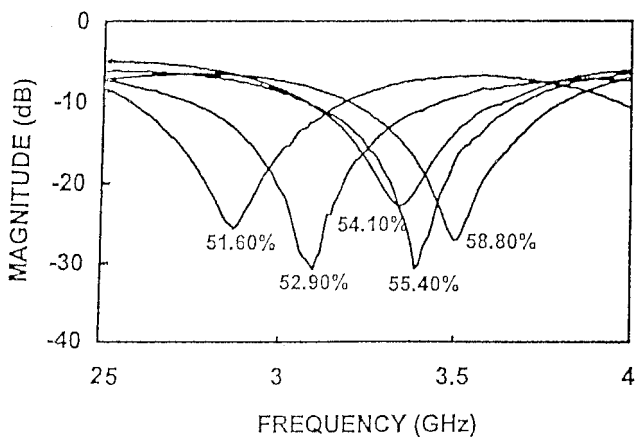

Figure 3. Return loss of open microstrip sensor immersed in curd cheese, as a function of frequency for various curd moisture content [35].

presented as a function of frequency for various moisture contents in cheese.

Sensitivity of moisture measurement can be significantly increased by using resonant structures as sensors. A microwave resonator is a metallic chamber resonating when the operating wavelength exactly matches its dimensions. Inserting a dielectric object into the cavity changes its electrical dimensions and the change can be correlated with the object permittivity and then with its moisture content. This principle has been used for moisture and mass determination of single kernels and seeds [36], as well as in bulk materials up to 5 liters in volume (sugar, pharmaceutical products, grain, cigarettes, margarine, etc.) measured in laboratory and industrial conditions [37,38]. A simple cylindrical resonator coupled through loops with external coaxial lines is shown in Figure 4. The output signal vs. frequency has the shape of a resonant curve, magnitude of which decreases for increasing moisture content in the material. The potential accuracy of frequency measurement has been used advantageously by coupling a pipe conducting liquid material (e.g., crude oil) into a microwave oscillator circuit in such a way that variation in water content changes the operating frequency of the system [39].

Two more interesting concepts should be mentioned here. One is using the effect of the relative permittivity of the medium on the cut-off wavelength of a waveguide (see Eq. (5)). For rectangular waveguide this can be written as $\lambda_c = c/2a\sqrt{\varepsilon'}$, where c is the speed of light and a the wider dimension of the waveguide [40]. Thus, when wet material is flowing through the waveguide, its moisture can be correlated to the attenuation measured along the waveguide. The second one is applying time-domain reflectometry (TDR), the technique well established for *in situ* determination of water content in soil with two-conductor line, to other granular materials such as grain [41]. These convenient two-wire sensors in combination with sharp pulse generating circuitry and precise time measuring devices can be a valuable complement to the family of high-frequency and microwave moisture meters.

4. Summary

This review does not pretend to be complete in covering all problems facing microwave aquametry. It was the intention of the author to show that microwave aquametry is a dynamic developing branch of metrology, full of practical potential and many needs for research in various fields of physics, chemistry and metrology. The truth is that a successful development requires not only an adequate microwave technology, but also specific

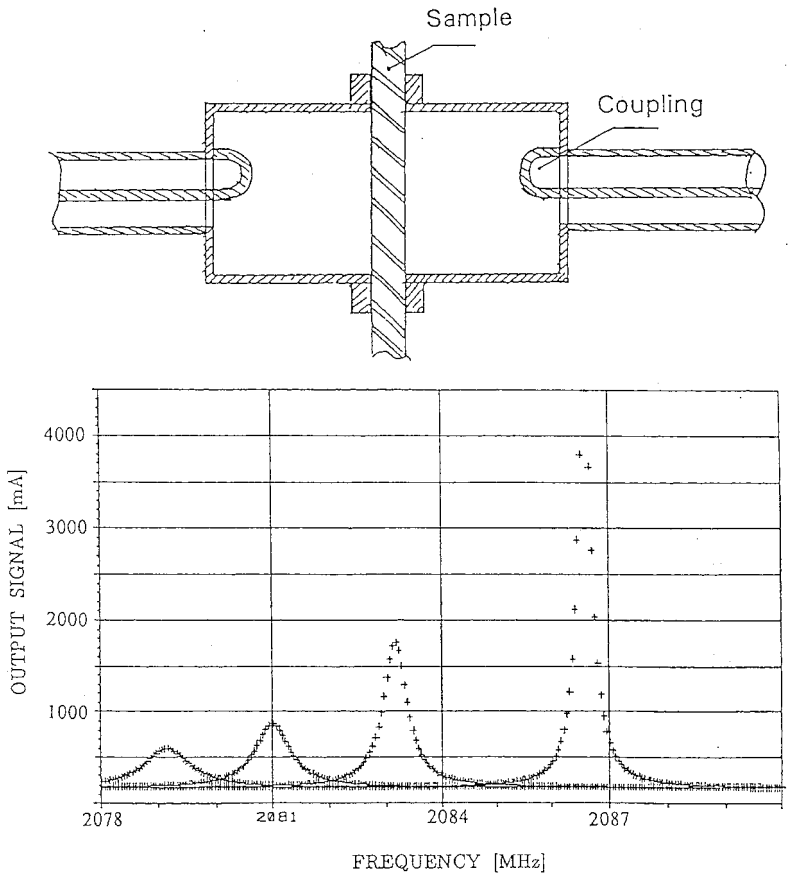

Figure 4. Cylindrical resonator and several resonant curves, with amplitude and frequency decreasing with higher moisture contents [37].

knowledge concerning the material of interest. While the microwave performance of a sensor can be expressed in terms of accuracy of amplitude and phase measurement, predicting the final accuracy in terms of moisture content dramatically depends on the properties of the material to be monitored and the particular details of the technological process. As a consequence, there is significant work to be done before making an effective microwave sensor for a given application.

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